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IN RE APPLICATION DOCKET NO.: 53647

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 DIETZEN ET AL.
 CONFIRMATION No.:
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 SERIAL No.
 10/516,921
 GROUP ART UNIT:
 1791

FILED: DECEMBER 07, 2004 EXAMINER: M. J. DANIELS

FOR: METHOD FOR PRODUCING EXPANDABLE POLYSTYRENE

Honorable Commissioner for Patents P.O. Box 1450 Alexandria, VA 22313-1450

DECLARATION UNDER 37 C.F.R. §1.132

I, Dr. Klaus Hahn, a citizen of the Federal Republic of Germany and residing at 67281 Kircheim, Im Bügen 9, Germany, hereby declare as follows:

I am a fully trained chemist, having studied Chemistry at the Universities of Clausthal and Marburg, Germany, from 1968 to 1974 and received a Diploma in Chemistry in 1974 from the University of Marburg.

I was awarded my doctors degree by the said University of Marburg in 1976;

I joined BASF SE, formerly named BASF Aktiengesellschaft, of 67056 Ludwigshafen, Germany, in 1977, and have since been working in the field of polymer foams including preparation and processing of expandable styrene polymers and I am therefore fully conversant with the technical field to which the invention disclosed and claimed in application Serial No. 10/516,921 belongs.

I worked as Research and Product Development Manager for thermoplastic foams, such as Styropor® (EPS) and are currently holding the position as Research Director for Thermoplastic Foams at the Polymer Research Laboratory at BASF SE.

Moreover, I am one on the inventors of the subject matter disclosed and claimed in application Serial No. 10/516,921, and I am familiar with the prosecution history of the application and with the prior art cited therein.

In the process disclosed and claimed the present application, high molecular weight styrene polymer is processed using a die plate having low diameter extrusion holes. Either one of these features would be expected to require a relatively higher extrusion temperature in the process according to Biglione (c.f., col. 3, indicated lines 28 to 47, of US 4,606,873), corresponding to the temperature of the polymer melt referenced in the claims of the application. However, the extrusion temperature is limited by polymer degradation and by decomposition of additives such as, e.g., flame retardants. The process disclosed and claimed in the present application ameliorates and overcomes the respective problems by heating the die plate to a temperature of from 20 to 100°C above the temperature of the blowing agent-containing styrene polymer melt. This measure allows that the extrusion temperature_of the blowing agent-containing styrene polymer melt can be kept lower.

Heating the die-plate not only prevents the formation of polymer deposits in the die-plate, and thereby assures long operation times for the granulation of high molecular weight styrene polymers, but also leads to small granule sizes of the expandable styrene polymer because of low die swell. This effect is, e.g., addressed on page 4, indicated lines 27 to 39, of the application and is also illustrated in Example 2 on page 7, indicated lines 10 to 27, of the application.

In particular, Example 2 demonstrates that it is possible to produce granules having a diameter of 0.60 mm or less, at a constant die exit diameter (D) of 0.4 mm and at a constant melt temperature of 200°C, when the temperature of the die-plate is increased to 220°C or above.

To further illustrate the unexpected advantages achieved by the process disclosed and claimed in application, I have conceived supplemental investigations. The tests were carried out by me, or under my direct supervision and in accordance with my instructions as specified below:

Example 2A:

The procedure of Example 2 on page 7 of the application was repeated with the difference that the blowing agent-containing polystyrene melt (6%by weight of n-pentane) was conveyed at a throughput of 110 kg/h (instead of 100 kg/h) through a die-plate having 300 holes (diameter at the die exit (D) 0.4 mm). The melt temperature was 200°C, and the die-plate temperature was 220°C.

The expandable polystyrene granules obtained in this manner had a uniform granule diameter of 0.65 mm. The molecular weight $M_{\rm w}$ of the expandable polystyrene was 241'000 g/mol after extrusion.

The granules were pre-formed to a density of 15 g/l. The cell number of the pre-formed granules was 4.4 cells/mm.

A comparison of Example 2A and Example 2 on page 7 of the application which differed in the throughput of the styrene polymer and in which the die-plate temperature was identical with the temperature of the polymer melt (cf., row 3 of the table on page 7 of the application) shows that the procedure disclosed and claimed in the application surprisingly allows for a more economic production of expandable styrene polymer particles of equal size.

Additionally, and to further illustrate the unexpected advantages which are achieved by the process disclosed and claimed in the application, I have conceived investigations into the demolding time, the shrinkage and the surface structure of sample moldings obtained using the expandable styrene polymer granules obtained in accordance with Example 2 of the application and in accordance with Example 2A described above.

Again, the tests were carried out by me, or under my direct supervision and in accordance with my instructions as specified below:

Determination of demolding time, shrinkage and surface structure

The expandable polystyrene granules obtained according to Examples 2 and 2A were pre-foamed to foam particles having a density of 15 g/l using steam. After 12 hours storage the foam particles were welded into foam moldings in a commercial mold (0.5 m X 1 m X 2 m) using steam blast at 0.6 bar pressure (1.6 bar absolute). The time until the pressure dropped to 0.05 bar (1.05 bar absolute) was measured and is indicated as demolding time.

In general, it is preferable that the demolding time be as short as possible, because a short demolding time allows a higher throughput and, thus, is advantageous because it provides for a more economic processing.

After 24 hours storage at room temperature (23°C) the foam moldings so obtained were cut into plates of 100 cm X 50 cm X 5 cm dimension. Shrinkage of the plates began immediately after cutting, and this time was defined as "zero." After cutting, each of the plates out of the middle of foam moldings were stored for 28 days at room temperature (23°C).

The change in the length of 5 individual plates was measured with an accuracy of 0.01 mm and the arithmetic average of these 5 plates was noted as degree of shrinkage in percent.

In general, it is preferable that the degree of shrinkage be as low as possible. A high degree of shrinkage of the plates can lead to damages in building insulation such as cracking in the plaster. In particular, the degree of shrinkage should be below 0.2%.

The surface structure of the plates was determined visually after storage and shrinkage. In general, an uneven matt surface of a foam plate indicates that coarse-celled foam particles were welded, whereas an even glossy surface indicates fine-celled foam particles were welded.

The data pertaining to die-plate temperature, granule diameter, throughput, demolding time, shrinkage and surface structure are compiled in the following table; the melt temperature was in each case 200°C:

No.	Die-Plate Temperature [°C]	Granule Diameter [mm]	Throughput [kg/h]	Demolding Time [sec]	Shrinkage [%]	Surface Structure
2.1	180	0.80	100	n / d	n/d	uneven, matt
2.2	200	0.65	100	89	0.23	uneven, matt
2.3	220	0.60	100	65	0.18	even, glossy
2.4	240	0.55	100	61	0.14	even, glossy
2.A	220	0.65	110	71	0.185	even, glossy

n/d = not determined

The granules obtained in the experiments in which the temperature of die-plate was the same as, or below, the temperature of the blowing agent-containing polymer melt, i.e., Examples Nos. 2.1 and 2.2, yielded plates having an uneven, matt surface due to a different cell structure and incomplete welding of the foam particles. Additionally, the demolding time and the shrinkage of the plates produced from the granules obtained in the experiment in which the temperature of die-plate was the same as the temperature of the blowing agent-containing polymer melt, i.e., Example No. 2.2, was significantly higher than the demolding time and shrinkage observed when granules were employed which had been obtained in experiments in which the die-plate temperature was higher than the temperature of the blowing agent-containing polymer melt.

The data in particular illustrate that the expandable polystyrene polymers which are obtained in accordance with the process disclosed and claimed in the application surprisingly are advantageous with a view to the quality of the foamed products, as evidenced by the

lowered degree of shrinkage and by the surface structure. The data also illustrate that the expandable polystyrene polymers which are obtained in accordance with the process disclosed and claimed in the application surprisingly allow for a more economic processing, as evidenced by the demolding time and the throughput.

I further declare that all statements made herein of my own knowledge are true and that all statements made on information or belief are believed to be true; and further that these statements are made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 101 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Signed at 67056 Ludwigshafen, this 19th day of January 2010.

(Signature of Declarant)